

A TFS-based Technique for Measuring Particle Size Distribution and Concentration in Flowing Suspensions

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Abstract

The intensity of a laser beam transmitted through a flowing suspension shows some fluctuations, due to the random discrete distribution of particles in the cuvette. The relationship was theoretically investigated among these temporal and spatial fluctuation signals, particle size distributions(PSD) and particle concentration by means of transmission fluctuation spectrometry(TFS) technique. Three different samples(glass beads, silicon carbide and large opaque particles) and a narrow focussed Gaussian beam of the TEM₀₀ mode He-Ne laser($\lambda = 632.8nm$) were used in this study. Some measurements on monodisperse and bi-disperse flowing suspensions were performed at low to medium volume concentration(up to about 15%). Some results on PSD and concentration are reported. It is concluded that this TFS-based technique promises to be applied in the on-line measurement and process control, due to the flow-through nature in the measurement.

Introduction

Particle size distribution(PSD) and particle concentration are two important parameters, when we analyze and control the quality of the products in industry or in the laboratory, such as measurement of particle concentration in two-phase flows, estimation of suspended particle content, control of crystal size, monitoring drinking water quality, and so on. An optical measurement method, based on the detection of the turbidity fluctuations through the particle suspensions, has been applied to determine mean particle diameter and concentration[1-2], by analyzing the root mean square values of the intensity fluctuations.

In some circumstances, it is required that PSD and particle concentration should be measured simultaneously. On the basis of statistical properties of transmission fluctuation signals, a new optical measurement method called transmission fluctuation spectrometry(TFS) has recently been studied for particle size analysis[3-7]. On the assumption that particles are opaque to the incident beam which can be infinitesimally thin or has a uniform or Gaussian profile, TFS was theoretically described with the expectancy of transmission square(ETS), whereby temporal averaging and/or spatial averaging were applied to measure the transmission fluctuations. Later, TFS was further investigated in the spatial domain theoretically and by numerical simulation[8], using dual beams and varying their distance, independent of the flow velocity.

In this study, theoretical deduction of correlation or the expectancy of transmission product(ETP) is described. A transition spectrum obtained from the transmission fluctuations is applied to extract the useful information of PSD and particle concentration in the flowing suspensions. In addition, a laboratory optical measurement system is given, which enables

simultaneous measurement of PSD and concentration. Subsequently detailed measurement results are presented.

Theory and Data Analysis

As the particle suspension flows through the cuvette at a flow velocity of v , the transmitted light intensity shows some temporal fluctuations, due to the random discrete distribution of particles. The transmission of light is the ratio of the intensity of transmitted light in the presence of particles to that in the absence of particles. These transmission fluctuation signals are related to the particle size x , particle concentration C_V , particle extinction efficiency K_{ext} and beam diameter D as well. For simplification, it is assumed that the particles in the flowing suspensions are opaque or perfectly absorbent to the incident beam, i.e. particle extinction efficiency equals 1.0. For a monodisperse monolayer with a monolayer density $\beta = P \cdot C_V$, where $P \geq 1.5$ is the structure parameter, the average transmission or the expectancy of transmission(ET) is $1 - \beta$. The monolayer is illuminated by a Gaussian beam which has the normalized beam function B_D .

$$B_D = \frac{8}{\pi D^2} \cdot e^{-\frac{8(\vec{r}-\vec{r}_0)^2}{D^2}} \quad (1)$$

where \vec{r}_0 is the coordinates of the beam center in the monolayer at starting time(see Fig.1).

The transmission $T_{1,ML,D}$ of the beam through the monolayer is the convolution of the transparency function T_{ML} of the monolayer at the beam center coordinate \vec{r}_0 with the normalized beam function B_D .

$$T_{1,ML,D}(\vec{r}_0) = (T_{ML} \otimes B_D)_{\vec{r}_0} \quad (2)$$

Due to the flow-through velocity of the particle suspensions, in a period of time τ , the transmission $T_{2,ML,D}$ of the beam through the monolayer can also be described by the convolution of the transparency function T_{ML} of the monolayer at the beam center coordinate $\vec{r}_0 + v\tau \cdot \vec{j}$ with the normalized beam function B_D .

$$T_{2,ML,D}(\vec{r}_0 + v\tau \cdot \vec{j}) = (T_{ML} \otimes B_D)_{\vec{r}_0 + v\tau \cdot \vec{j}} \quad (3)$$

where \vec{j} is the unit vector in the direction of flow velocity of the particle suspension.

The correlation or the expectancy of transmission product(ETP) through the monolayer with an area A is defined as

$$ETP = \lim_{A \rightarrow \infty} \frac{1}{A} \iint_{\vec{r}_0 \in A} T_{1,ML,D}(\vec{r}_0) \cdot T_{2,ML,D}(\vec{r}_0 + v\tau \cdot \vec{j}) \cdot d\vec{r}_0 \quad (4)$$

By using Fourier transform similar to the deduction of ETP on a spatial scale[8], ETP on a time scale is given by

$$ETP_i = 1 - \beta \cdot [2 - \chi_i(\zeta, \Lambda)] + O(\beta^2) \quad (5)$$

$$\approx 1 - \beta \cdot [2 - \chi_i(\zeta, \Lambda)]$$

$$\chi_i(\zeta, \Lambda) = \int_{u=0}^{+\infty} J_0(2u\zeta) \cdot e^{-\frac{u^2 \Lambda_i^2}{4}} \cdot \frac{2J_1^2(u)}{u} \cdot du \quad (6)$$

where $\zeta = v\tau/x$ is dimensionless correlation time with autocorrelation technique, $\Lambda = D/x$ is

dimensionless beam diameter, $J_0(\cdot)$, $J_1(\cdot)$ are the 0th and 1st order Bessel functions. For low particle concentration, the second order term $O(\beta^2)$ of monolayer density in Eq.(5) is omitted. The subscript i refers to the i^{th} monolayer in the flowing suspensions. Thus by the operation of logarithm, Eq.(5) can be approximated as $\ln ETP = -\beta(2 - \chi)$. Fig.2 illustrates the LnETP for a monodisperse suspension (glass beads 480-520 μm , $C_V=0.98\%$). The position of turning point in the LnETP curve gives the particle size, and the height gives the monolayer density β or particle volume concentration C_V .

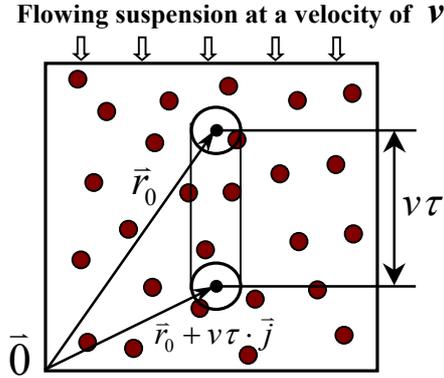


Fig. 1: Monodisperse Flowing Suspension

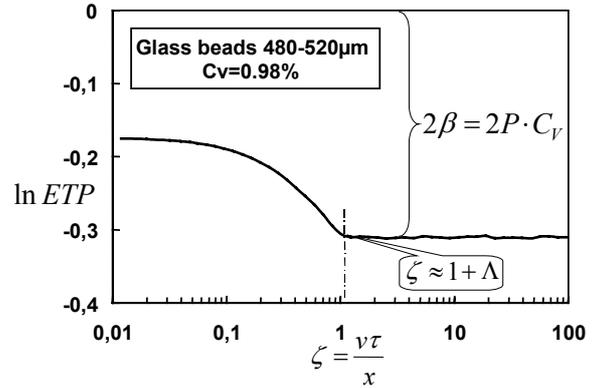


Fig. 2: LnETP for Monodisperse Suspension

In the practical application, the beam passes through the cuvette with a certain optical path length or thickness ΔZ greater than the monolayer thickness $\Delta Z_{ML} = P \cdot x / 1.5$. On the basis of layer model[9], a three-dimensional flowing suspension can be modelled as a pile of monolayers, which are statistically independent of each other. In the low particle concentration limit, the total ETP is the product of ETPs through individual monolayers. Meanwhile, a polydisperse suspension can also be modelled as a group of monodisperse suspensions. Thus, the total ETP through a flowing suspension illuminated by a focussed Gaussian beam of the TEM₀₀ mode He-Ne laser ($\lambda = 632.8\text{nm}$) is given by

$$ETP_{TOT} = \prod_{i=1}^{N_{ML}} ETP_i \quad (7)$$

where N_{ML} is the number of monolayers in the flowing suspension.

By combining Eq.(5) and Eq.(7), we can obtain, for a polydisperse suspension,

$$\ln ETP_{TOT} = -\sum_k \left[2 - \frac{1}{\Delta Z} \int_{z=-\Delta Z/2}^{\Delta Z/2} \chi(\zeta, \Lambda(x_k, z)) \cdot dz \right] \cdot \frac{1.5}{x_k} \cdot C_V(x_k) \cdot \Delta Z \quad (8)$$

$$\text{with } \chi(\zeta, \Lambda(x_k, z)) = \int_{u=0}^{+\infty} J_0(2u\zeta) \cdot e^{-\frac{u^2 \Lambda^2(x_k, z)}{4}} \cdot \frac{2J_1^2(u)}{u} \cdot du \text{ and } \Lambda(x_k, z) = \frac{2\omega_0}{x_k} \sqrt{1 + \left[\frac{z\lambda}{\pi\omega_0^2} \right]^2}.$$

where ω_0 is waist radius of Gaussian beam, and x_k is the particle diameter of the k^{th} size class. Eq.(8) means that the logarithm of total ETP is the linear superposition of all the monodisperse curves.

In the real measurements, ETP and ET can be obtained directly from the transmission fluctuations produced by the particles in the flowing suspensions. Therefore, for practical purposes, the transition function H of ETP is defined as the ratio of the logarithm of total ETP to the logarithm of total ET.

$$H = \frac{\ln ETP_{TOT}}{\ln ET_{TOT}} \quad (9)$$

The transmission fluctuation signals can be acquired and digitized with a high-speed data acquisition card. These discrete data are further analyzed by a personal computer. By changing the autocorrelation time τ , which corresponds to a group of dimensionless correlation time $\zeta = v\tau/x$, a transition spectrum can be obtained. Eq.(8) shows the relationship among transmission fluctuation signals, particle volume concentration C_V and particle size x_k . Therefore, using a proper inversion method, the information of PSD and concentration can be retrieved.

Experimental Method

Three different samples(glass beads, silicon carbide and large opaque particles) and two different cuvettes were used in this study. Fig.3 illustrates the optical measurement setup in the experiment. A Gaussian beam of the TEM₀₀ mode He-Ne laser($\lambda = 632.8nm$) was used as illuminating light. The laser beam was expanded and collimated with a beam expander, and then focussed with a convex lens. The beam waist(about 12 μm) lay in the middle of the cuvette. For the measurement on glass beads and silicon carbide, a cuvette with a pathlength of 5mm was used. The suspension was recirculated through the cuvette at a flow rate of about 1.98m/s using a hose pump driven by a motor. The total volume of flowing suspension in the cycling pipe was about 1210ml. The rotative velocity of the pump was monitored and controlled to avoid suction of the air bubbles in the cycling pipe. For the measurement on large opaque particles, made of alginate sodium, the other cuvette with a pathlength of 10.5mm was applied. In order to avoid deforming or destroying the large alginate particles during the recirculation through the hose pump, compressed air was used to pump the particle suspension with a total volume of about 550ml at a flow rate of about 0.184m/s. The pressure of the compressed air was controlled with a barometer. The intensity of the laser beam transmitted through the flowing suspension was focussed onto a photodiode(PBX65) receiver with a small receiving aperture angle of about 0.04degrees. The transmission fluctuation signals from the photodiode were amplified and sent to the data acquisition card. In this study, a high time-resolution digital oscilloscope(DL708, made by YAKOGAWA company) was applied to acquire the real-time transmission fluctuation signals. The acquired discrete data were transmitted to computer for further data analysis.

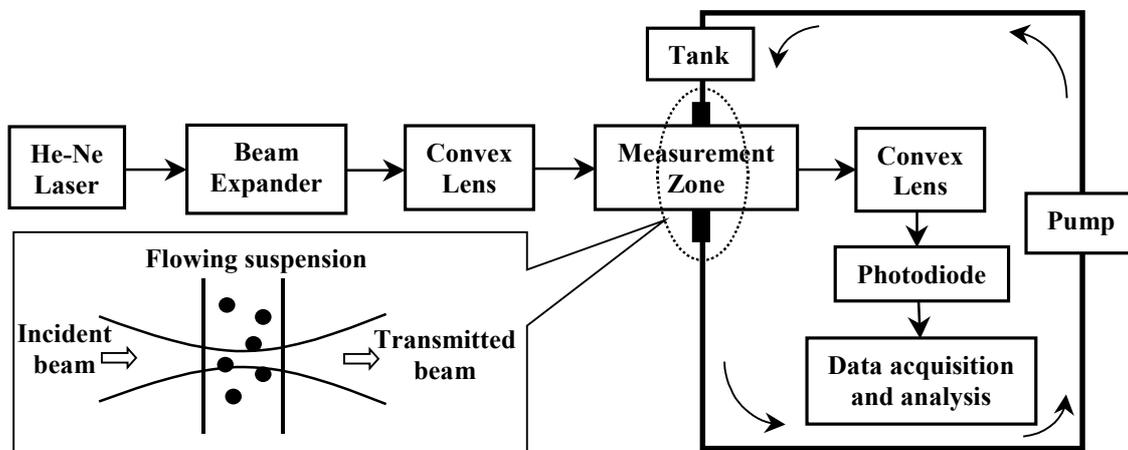


Fig. 3: Optical Measurement Setup

Results and Discussion

Three different samples (glass beads, silicon carbide, large alginate opaque particles) with different nominal diameters were measured at different volume concentrations in this experiment. In the measurements on glass beads (Gls) and Silicon carbides (SiC), the thickness ΔZ of the cuvette was about 5mm and the corresponding equivalent average parallel beam diameter through this cuvette was about 50 μm . Similarly, the equivalent average parallel beam diameter was about 93 μm for $\Delta Z = 10.5\text{mm}$ in the measurement on large alginate opaque particles (Alg). SiC samples are perfectly absorbent and in this case the extinction efficiency of SiC larger than beam diameter is expected to be 1.0. Actually, the sphericity of SiC has an effect on the extinction efficiency and hence makes its extinction efficiency larger than 1.0. For larger transparent particles, such as glass beads used in this study, the transmitted light was diverted to sufficiently large angles so that it can't enter the receiving aperture. Only when the particle is located very close to the beam waist, could part of the refracted light be received [10]. This seldom happens in the measurement. Therefore, in this case, while they are not absorbent, there is an equivalent effect, and the extinction efficiency also equals 1.0. However, for the particles smaller than or close to the beam diameter but much greater than the wavelength of incident beam, extinction efficiency is expected to be 2.0. In reality, due to the influence of diffracted light, extinction efficiency decreases and ranges between 1.0 and 2.0 [10].

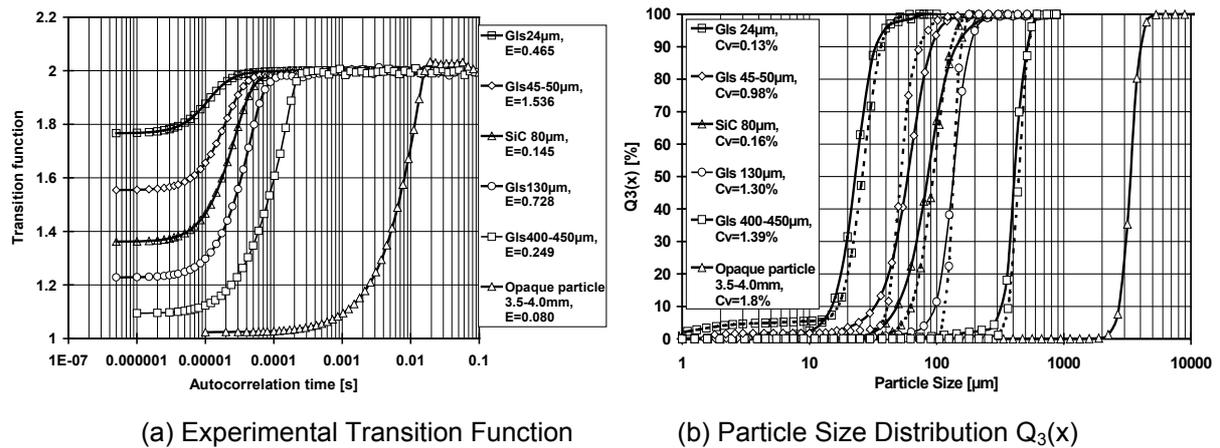


Fig. 4: Experimental Transition Function and Particle Size Distribution of Monodisperse Suspension

The acquired temporal and spatial fluctuation signals were analyzed by using TFS autocorrelation technique (TFS-AC) [11]. Experimental transition functions of different samples at low particle concentrations were obtained, seen in Fig. 4a. The smaller the particle, the smaller the step height of its transition function. This was due to the spatial average over the beam cross-section, and hence the transmission fluctuation signals produced by small particles became weaker than those produced by larger ones. In order to extract the information of PSD and concentration with Eq. (8), a modified Chahine inversion algorithm [12] was applied. In the inversion procedure, it was assumed that all the particles were spherical and the extinction efficiency of particles was 1.0. The corresponding particle size distributions $Q_3(x)$ obtained by TFS technique were plotted with dotted solid lines in Fig. 4b. The median values x_{50} agreed with the nominal diameters of the samples.

For the above monodisperse samples less than 1000 μm , they were also measured with HELOS instrument (made by SYMPATEC company), based on the laser diffraction. The

results $Q_3(x)$ obtained by HELOS were also plotted with dotted dash lines in Fig.4b. It was found that the PSDs by TFS technique were consistent with those by HELOS. HELOS gave a slightly higher resolution. However, for smaller particles, such as glass beads $24\mu\text{m}$ and glass beads $45\text{-}50\mu\text{m}$ in Fig.4b, Fraunhofer results by HELOS showed an artefact quantity of small particles between $1\mu\text{m}$ and $10\mu\text{m}$. It could be explained that the laser light passing through the glass beads formed a constructive interference with the light diffracted from the edge of the glass beads. These interference signals were recorded by outer ring detectors. The Fraunhofer approximation didn't model transparency and hence interpreted these interference signals as the presence of a quantity of small particles.

Fig.5 illustrates the comparison between particle volume concentrations measured by TFS technique and those by mass. It was found that, over a wide range of particle concentrations and for larger spherical particles (e.g. glass beads and large alginate opaque particles used in this experiment), almost all the experimental data fitted to the results by mass very well up to about 15%. However, for nonspherical SiC samples (e.g. SiC $80\mu\text{m}$, plotted with hollow dots o), the measured results showed an overestimation because of the effect of particle sphericity. In addition, for the measurements on small spherical particles less than beam diameter (e.g. glass beads $1.68\text{-}15.7\mu\text{m}$, plotted with crosses +), there also appeared an overestimation, which resulted from the effect of particle extinction efficiency (greater than 1.0), as discussed above.

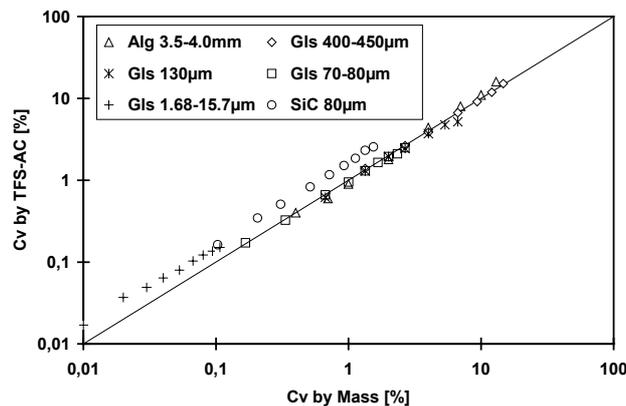
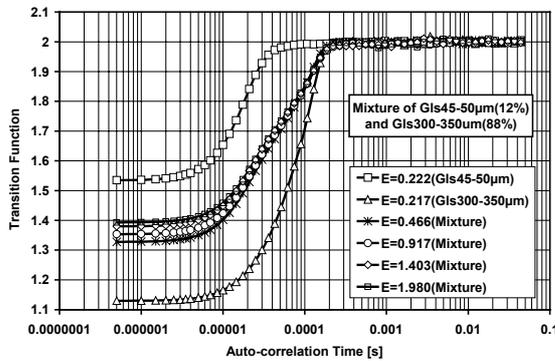


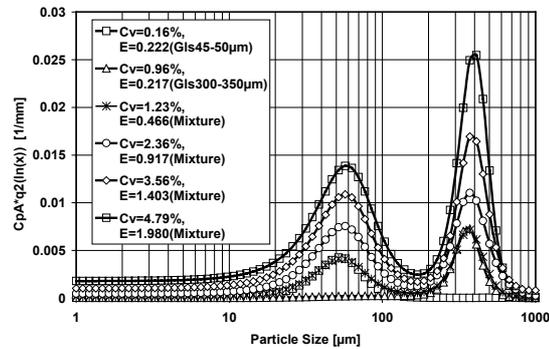
Fig. 5: Particle Volume Concentrations Measured by TFS Technique and by Mass

Some measurements on bidisperse suspensions were also carried out. The flowing bidisperse suspensions were prepared by mixing glass beads $45\text{-}50\mu\text{m}$ and glass beads $300\text{-}350\mu\text{m}$ at a volume concentration ratio (12% : 88%). Before the measurement on the mixture, these two monodisperse samples were measured respectively. The experimental transition functions of Gls $45\text{-}50\mu\text{m}$ and Gls $300\text{-}350\mu\text{m}$, corresponding to the extinction $E=0.222$ and $E=0.217$ respectively, were plotted in Fig.6a. Then these two samples were mixed and measured. It can be seen that the transition function of this flowing bidisperse suspension ($E=0.466$) was almost the linear superposition of these two monodisperse suspensions. In the above section, our theoretical description is also based on the assumption that the steric interactions between particles in the dilute polydisperse particle systems are negligible and a polydisperse suspension can be modelled as a group of monodisperse suspensions. The total extinction is the contributions from these individual monodisperse suspensions. It can be seen from Fig.6b that the inverted particle projected area concentration of the bidisperse suspension ($E=0.466$, plotted with stars *) appears to be the sum of the projected area concentration of Gls $45\text{-}50\mu\text{m}$ ($E=0.222$, plotted with hollow dots \square) and that of Gls $300\text{-}350\mu\text{m}$ ($E=0.217$, plotted with hollow dots Δ). By increasing the amount of these two samples in proportion, the bidisperse suspensions were measured at different

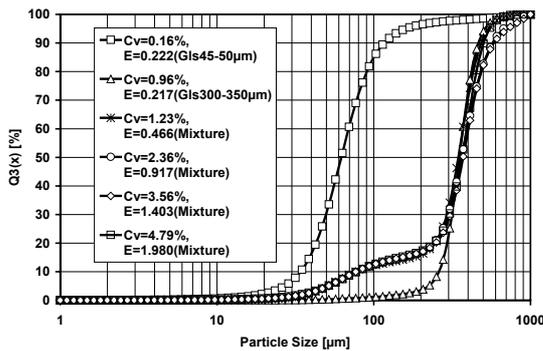
concentrations. There existed a gradual shift of transition functions with increasing extinction E or particle concentration, seen in Fig.6a. The inverted particle projected area concentrations and PSDs were illustrated in Fig.6b and Fig.6c, respectively. But the concentration effect visible on cumulative volume distribution $Q_3(x)$ was very small. Fig.6d showed that the particle volume concentrations measured by TFS technique agreed with those by mass.



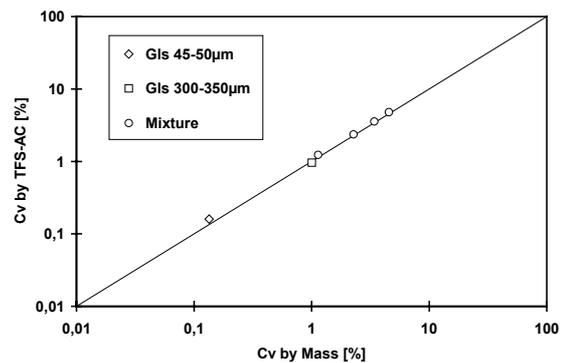
(a) Experimental Transition Function



(b) Projected area concentration $C_{PA} \cdot q_2(\ln(x))$



(c) Particle Size Distribution $Q_3(x)$



(d) C_v Measured by TFS Technique and by Mass

Fig. 6: Experimental Results on Bidisperse Suspension

The above measurements were carried out with a single beam. By applying dual beams passing through the fluid in parallel with a certain geometric distance in the flow direction, the flow velocity could be measured through the cross-correlation analyses on the transmission fluctuation signals from these two beams. In addition, if the dimension of the flowing suspension is much larger than particle diameter and beam diameter, the locally resolving measurement on PSD and particle concentration at different positions in the flowing suspension could also be realized by moving the position of effective measurement zone in the flowing suspension. These further investigations of TFS technique would be interesting and help to monitor the flow structures, such as in fluidized beds, pneumatic transportation and so on.

Conclusion

A TFS-based technique was introduced to measure particle concentration and particle size distribution(PSD) in the flowing suspensions simultaneously, described in terms of the expectancy of transmission product(ETP) theoretically. A focussed Gaussian beam of the

TEM₀₀ mode He-Ne laser ($\lambda = 632.8nm$) was used in the real measurement. The temporal and spatial transmission fluctuation signals produced by the particles in the flowing suspensions were detected for particle size analysis. Some measurements on monodisperse and bidisperse suspensions were carried out at low to medium volume concentrations. The PSDs obtained by TFS technique were in a good agreement with those by HELOS instrument. For the spherical particles larger than beam diameter, measurement results on particle volume concentration by TFS technique fitted very well to those by mass up to about 15%. However, the experimental results displayed that there appeared an overestimation of particle volume concentration for the non-spherical particles (SiC samples) and smaller particles (less than beam diameter), due to the influence of particle sphericity and particle extinction efficiency, respectively.

Because of the simple experimental setup and the flowing-through nature in the measurement, on-line measurement and process control could be realized with this technique.

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